
पेट्रोलियम और उसके उत्पादों के लिए परीक्षण के तरीके

[पी : 141] हीलियम विधि द्वारा कैल्क्लाइंड पेट्रोलियम
कोक के स्पष्ट घनत्व के निर्धारण
(पहला पुनरीक्षण)

Methods of Test for Petroleum and Its Products

[P : 141] Determination of Apparent Density of Calcined
Petroleum Coke by Helium Method

(*First Revision*)

ICS 75.160.10

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भारतीय मानक ब्यूरो

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FOREWORD

This Indian Standard [P : 141] (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Sampling and Test for Petroleum, Petroleum Products, Gaseous Fuels and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The real density of calcined petroleum coke directly influences the physical and chemical properties of the manufactured carbon and graphite artifacts for which it is used. Density, therefore, is a major quality specification of calcined petroleum coke and is used as a control in coke calcination. The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

The mass per unit volume (or the weight per unit volume) of a material, including the voids which are inherent in the material. Apparent density reflects the ability to provide proper structural support. It is also utilized to convert between volume and weight. This measurement of apparent density can help solve issues that result from poor function. For instance, high levels of high apparent density indicate poor compaction and porosity.

This standard was initially published in 1992 and has a test method using Beckman apparatus. In this revision, considerable assistance has been derived from ASTM D 2638 “Standard Test Method for Real Density of Calcined Petroleum Coke by Helium Pycnometer”.

The following Indian Standards, may be referred to for sampling of coal and coke:

IS 436 (Part 1/Sec 1) : 1964	Methods for sampling of coal and coke: Part 1 Sampling of coal, Section 1 Manual sampling
IS 16143 (Part 4) : 2014/ ISO 13909-4 : 2001	Hard coal and coke — Mechanical sampling: Part 4 Coal — Preparation of test samples
IS 16143 (Part 6) : 2014/ ISO 13909-6 : 2001	Hard coal and coke — Mechanical sampling: Part 6 Coke — Preparation of test samples
IS 16143 (Part 7) : 2014/ ISO 13909-7 : 2001	Hard coal and coke — Mechanical sampling: Part 7 Methods for determining the precision of sampling, sample preparation and testing
IS 16143 (Part 8) : 2014/ ISO 13909-8 : 2001	Hard coal and coke — Mechanical sampling: Part 8 Methods of testing for bias

This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 141] DETERMINATION OF APPARENT DENSITY OF CALCINED PETROLEUM COKE BY HELIUM METHOD

(*First Revision*)

1 SCOPE

This test method covers the determination of the real density (RD) of calcined petroleum coke. Real density, by definition is obtained when the particle size of the specimen is smaller than 75 microns (No. 200 Sieve).

2 REFERENCE

The Indian Standard listed below contains provision which, through reference in text, constitutes provision of this standard. At the time of publication, the edition indicated was valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below.

<i>IS No.</i>	<i>Title</i>
1448 [P : 130] : 1988	Methods of test for petroleum and its products: [P : 130] Determination of vibrated bulk density of calcined petroleum coke

3 TERMINOLOGY

Following definition shall apply while using this standard:

3.1 Calcined Petroleum Coke — Petroleum coke that has been thermally treated to drive off the volatile matter and to develop crystalline structure.

3.2 Petroleum Coke — A solid, carbonaceous residue produced by thermal decomposition of heavy petroleum fractions or cracked stocks, or both.

3.3 Bulk Density — The mass of the particles divided by the volume they occupy which includes the space between the particles (*see* IS 1448 [P : 130]).

3.4 Real Density (RD) (also be referred to as true specific gravity) — The mass divided by the volume occupied by the material excluding pores and voids. It is required, therefore, that voids in the coke be eliminated and that pores in the material be filled by the fluid being displaced. This requirement is met for the purposes of this test method by reducing the coke particles to a size smaller than 75 microns.

NOTE — The density of particles larger than 75 microns up to the largest that can be put into the helium pycnometer can also be determined, but must be designated as particle density (PD). The precision data obtained for RD may not be applicable to PD.

4 OUTLINE OF TEST METHOD

A representative sample of calcined petroleum coke is dried and ground to pass a 75 micron (200 mesh) screen. The mass of the sample is determined directly and the volume derived by the volume of helium displaced when the sample is introduced into a helium pycnometer. The ratio of the mass of the sample to the volume is reported as the real density.

5 INTERFERENCES

Oil or other de-dust material sprayed on calcined petroleum coke to control dust will interfere with the determination of real density so the oil must be removed before reducing the sample to 75 microns.

6 APPARATUS

6.1 Analytical Balance, accurate to ± 0.1 mg.

6.2 Desiccator

6.3 Drying Oven, preferably a vacuum oven, for temperature to 120°C.

6.4 Helium Pycnometer

6.5 Jaw Crusher and Roll Crusher, other style crushers which allow control over particle size without contamination are acceptable.

6.6 Riffles, with hoppers and closures.

6.7 Wire Sieve, 75 microns (No. 200 mesh)

7 REAGENTS

Helium, 99.9 percent

8 SAMPLE PREPARATION

8.1 Reduce and divide the gross sample to obtain a laboratory analysis sample.

8.2 Divide, by riffing, a minimum of 100 g from the laboratory analysis sample.

NOTE — The amount of coke required will be dictated by the operating instructions supplied by the helium pycnometer manufacturer. However, to assure that the test sample is representative of the entire sample, a minimum sample size of 100 g is required.

8.3 When dedusting oil is present it must be removed prior to further sizing.

8.4 Crush 100 g of the test sample so that the entire sample will pass through a 75 micron (No. 200) sieve. Dry the crushed sample in a drying oven at $115 \pm 5^\circ\text{C}$ to constant mass (approximately 8 h). Cool in a desiccator.

9 PROCEDURE

9.1 Place 5 to 100 g (80 to 90 percent of the maximum capacity of the particular holder being used) of the prepared crushed, sized and dried test sample into the specimen holder provided with the instrument. Weigh to the nearest milligram.

9.2 Place the pre-weighed test sample and specimen holder into the sample chamber and evacuate.

9.3 Connect, either manually or automatically, the control chamber, containing helium at a higher pressure than the sample chamber, with the sample chamber. Determine the final equilibrated pressure.

9.4 The volume of the calcined petroleum coke sample is calculated from the known volume of the two chambers, the absolute pressures in each chamber prior to connection and the final pressure after the chambers have been connected.

9.5 Record the final sample volume or density when the pycnometer has reached equilibrium.

NOTE — This is a generic procedure which describes the basic steps involved to obtain a final result. For further specific details, the manufacturers' operate instructions are to be reviewed.

10 CALCULATION

Determine the real density of the sample in g/cm^3 .

$$\text{Density} = M/V$$

where

M = mass of sample, in g; and

V = volume of helium displaced, cm^3 .

11 REPORT

Report to the third decimal place the real density as calculated in 9.

12 PRECISION AND BIAS

Method precision to be established based on the round robin outcome.

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This Indian Standard has been developed from Doc No.: PCD 01 (2874).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi